National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 2063a

Microanalysis Thin Film Mg-Si-Ca-Fe

This Standard Reference Material (SRM) is intended for use in the standardization of chemical analysis by x-ray and energy loss spectrometry on the analytical electron microscope. This film may be used to determine relative sensitivity factors as described in many references [e.g., 1,2]. SRM 2063a consists of a mineral glass film that has been deposited onto a 20-nm thick carbon support film on a 3-mm diameter, copper transmission electron microscope grid. The thickness and density of the film have been determined and are presented as supplemental information.

The certified values for Mg, Si, Ca, Fe, and O, given below, are based on measurements made using several analytical techniques. The value for Ar in parenthesis is not certified but is given for information only.

Element	Certified Concentration % by weight[a]	Uncertainty % by weight[b]
Mg	7.97	0.34
Si	25.34	0.98
Ca	11.82	0.37
Fe	11.06	0.88
O	43.2	1.6
Ar	(0.4)	

[a] The certified value listed for an element is a weighted mean value pooling results from several analytical techniques [3,4].

[b] The stated uncertainty includes allowances for measurement imprecision, material variability, and differences among analytical methods. Each uncertainty is the sum of the half-width of a 95% prediction interval and an allowance for systematic error among the methods used. In the absence of systematic error, a 95% prediction interval predicts where the true concentrations of 95% of the samples of this SRM lie. [3,4].

The overall direction and coordination of the technical measurements leading to certification were under the direction of E.B. Steel and R.A. Velapoldi, Chief, NIST Surface and Microanalysis Science Division.

Statistical analysis of the certification data was provided by S.D. Leigh and S.B. Schiller of the Statistical Engineering Division.

The technical and support aspects involved in the certification and issuance of this Standard Reference Material were coordinated through the Standard Reference Materials Program by J.S. Kane.

Gaithersburg, MD 20899
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William P. Reed, Chief
Standard Reference Materials Program

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Use: The dark side of the copper grid contains the glass film supported by carbon. The dark side should face the x-ray detector for calibration procedures. High electron beam doses (above approximately 10^{-5} C cm^2) may cause instability in the chemical composition of the thin film. For this reason it is suggested that only defocussed beams or scanned area analyses be used on this standard and that the analyst test for beam damage by analyzing at several beam currents.

Preparation: The films were prepared by J.M. Phelps of the NIST Surface and Microanalysis Research Division using focussed argon ion beam sputtering from a glass target onto the copper support grids. The glass target was fabricated by D. Blackburn and D. Kauffman of the NIST Ceramics Division.

Chemical Analysis: Electron probe microanalysis, analytical electron microscopy, x-ray fluorescence, and/or time-of-flight secondary ion mass spectrometry were used to determine the chemical composition of the Mg, Si, Ca, Fe, and O in the thin film. The Ar content was calculated using a Monte Carlo program and the measured x-ray intensities from the films. Trace concentrations of Ni, Cr, and Mn were also observed in the films. The analysts were J.A. Bennett, J.M. Phelps, and E.B. Steel of the NIST Surface and Microanalysis Science Division.

Supplemental Information

The thickness of the films was measured by profilometry and was found to be 76 nm with 95% confidence limits of $\pm$ 4 nm. A density of 3.1 g/cm^3 with 95% confidence limits of $\pm$ 0.3 g/cm^3 was calculated from the measured thickness, area, and mass of the thin-film depositions. These values are not certified but are given for information only.

REFERENCES

[1] Cliff, G., and Lorimer, G.W., J. Micros, Vol. 110, p. 107, (1975).

[2] Joy, D.C., et al., eds., Principles of Analytical Electron Microscopy, Plenum Press, New York, (1986).

[3] Paule, R.C. and Mandel, J., Consensus Values and Weighting Factors, J. Res., Nat. Bur. Stand. (U.S.), 87, (5) 377-385 (Sept.-Oct., 1982).

[4] Schiller, S.B., and Eberhardt, K.R., Combining Data from Independent Chemical Analysis Methods, Spectrochim. Acta, 46B, 12, pp. 1607-1613, (1991).